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FAST-BURNING RATE/HIGH SLOPE PROPELLANT TECHNOLOGY PROGRAM

SECOND QUARTERLY PROGRESS REPORT

1 August–31 October 1970

(U)

by
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Aerojet General Corporation
for the
Propulsion Development Department

ABSTRACT. (U) This is the second quarterly progress report on work conducted to advance state-of-the-art with regard to formulation of practical fast-burning and high pressure-exponent propellants. Primary emphasis was directed toward optimization of the processing, mechanical, and ballistic properties.

(U) Approaches to improving propellant processability included variation of amount and type of plasticizer, use of blocked or hindered isocyanate curing agents, evaluation of epoxide curing agents and modification of R-45M prepolymer. The last approach was found to be by far the most effective, and satisfactory processability can be achieved with propellants meeting the desired ballistic and mechanical properties.

NAVAL WEAPONS CENTER
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W. J. Moran, RADM, USN ..... Commander

H. G. Wilson ..... Technical Director

FOREWORD

This second quarterly report describes progress during the period 1 August through 31 October 1970 on Navy Contract N00123-70-C-1457 to Aerojet General Corporation, Sacramento, California. This work is sponsored by the Naval Weapons Center (NWC), China Lake, Calif., and supported by the Naval Air Systems Command under AirTask A3303300/216B/IF 19332302.

F. M. Pickett of NWC is the technical coordinator and has reviewed this report for technical accuracy.

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CONTENTS

INTRODUCTION

(U) This is the second quarterly progress report presented to the U. S. Navy, Naval Weapons Center, China Lake, California, for work conducted under contract N00123-70-C-1457 for the period 1 August 1970 through 31 October 1970. A glossary of terms and abbreviations is provided.

OBJECTIVE

(U) The objective of this 10-month program is to advance the state-of-the-art with regard to formulation of practical fast-burning and high pressure-exponent propellants by expanding available technology.

(U) The research performed will provide the capability to formulate two propellants, hereinafter referred to as "A" and "B", with respective burning rates of 3.5 and 7.0 in/sec. at 2000 psia and a pressure exponent for both propellants of about 0.70.

(U) Both propellants will be formulated to deliver a specific impulse (I^{15}_{1000}) of at least 240 lbf-sec/lbm with a density of 0.063-0.065 lbs/in.^{3}. The study also includes the development of adequate mechanical properties to withstand the temperature range of -40 to 160°F. Other considerations are adequate processing, potlife (4 hrs. @ 135°F), thermal and aging stability and safety characteristics. Only composite propellants are being considered, with porous AP (PAP) and non-volatile ferrocene derivatives limited to the high burning rate propellant "B".

SUMMARY

(U) Primary emphasis during this report period was directed toward optimization of the processing, mechanical and ballistic properties of propellant "A". A lesser effort was expended on propellant "B" since progress made on "A" was readily applied towards the optimization of "B".

(U) Approaches to improving propellant processability included variation of amount and type of plasticizer, use of blocked or hindered isocyanate curing agents, evaluation of epoxide curing agents and modification of R-45M prepolymer. The last approach was found to be by far the most effective, and satisfactory processability can be achieved with propellants meeting the desired ballistic and mechanical properties. Selection of the final candidates has been postponed until the next report period due to a one-month program extension.

(U) The use of acetylacetone and trioctylphosphine oxide (TOPO) to, respectively, block and dimerize the TDI in situ by adding them to the propellant premix did not yield any increase in potlife. Dimerized TDI was prepared and used in several batches. An adequate potlife was obtained, but the cured propellant was too brittle. Further work with this material was discontinued.

(U) Mixed epoxy/isocyanate curing agents yielded some encouraging results in extending propellant potlife, but failed to provide satisfactory cures. In all, four diepoxides were evaluated and were found to either provide fair potlives and poor cures, or poor potlives and brittle cures. It was concluded, therefore, that the use of epoxides, alone or in combination with IPDI, do not provide significant benefits.

(U) The replacement of the Oronite 6 with IDP resulted in a somewhat more processable propellant. However, the cured propellant yielded poorer mechanical and ballistic properties than was achieved with Oronite 6 as the plasticizer. No further attempt was made to use IDP, therefore, because the processing improvement it provides is more than offset by its deterioration of other propellant properties.

(U) Using Oronite 6 as the plasticizer, an attempt was made to improve processing and thereby increase potlife by lowering the R-45M to stoichiometrically match the IPDI level, making up the difference with plasticizer. No significant gains in potlife were achieved, and both ballistic and mechanical properties suffered.

(U) The addition of aluminum near the end of the mix cycle rather than at the beginning was investigated to determine if its presence in the long interim mix cycle had any processing, mechanical property or ballistic effects. Minor effects were observed on ballistic and mechanical properties, but not on processing. A moderate improvement in processing, however, was observed when a previously unopened can of the current lot of R-45M was used. The improvement in processing was ascribed to less oxidative crosslinking in the unexposed R-45M.

(U) Lower ballistic solids were investigated as a means to improve both processing and mechanical properties. Although improvements were seen in these areas, the sacrifice in burning rate was too great to seriously consider this approach. A promising approach, however, towards improving propellant potlife was achieved by using a modified R-45M. Significant increases in potlife were achieved with little change in mechanical properties.

(U) A series of three batches were prepared varying the IPDI from 45-55-65 equivalents. Viscosity buildup and mechanical property data indicate proper IPDI equivalents lie between 55 and 60. However, higher equivalents levels of IPDI were required as the DEO level was dropped. This was shown in a series of three batches which were made

to further substantiate the need for and establish the desired levels of DEO and TEA in the formulation. The data indicate that the best overall properties are achieved with both present, and that the proper IPDI level lies between 65 and 70 equiv. in a system where the R-45M level has been raised by 10 equivalents.

(U) Two other methods of increasing the burning rate of propellant "A" were evaluated. The first method consisted of adding a fluorine containing plasticizer (ester of 2-ethylhexanoic acid with 1,1,9-trihydrohexadecafluoro-1-nonanol) to the propellant replacing half of the plasticizer. The resulting composition processed with difficulty and cured to a brittle propellant which could not be tested for burning rate. The second method involved preparing an additional batch of propellant "A" incorporating two percent of the ground ferrocene-formaldehyde polymer previously evaluated at 0.5% level. No significant burning rate increase was achieved, and processability was severely curtailed.

(U) Several significant improvements were made in increasing the burning rate of propellant "A". The replacement of the iron oxide previously used by a crystalline red iron oxide provided by Naval Weapons Center resulted in a 7% increase in burning rate at 2000 psia. A 5% increase was realized by the replacement of 19% MA AP (10.) with 3. UFAP and another 7% increase by doubling the mix cycle to 4 hours. By combining these factors, a solid strand burning rate of 3.62 in./sec at 2000 psia was achieved. A repeat batch was made to confirm this data which yielded a lower burning rate of 3.4 in/sec at 2000 psia. The reason for the rate drop is unknown.

(U) Replacing the old Fe_2O_3 and Silon-S combination entirely with the crystalline Red Fe_2O_3 gave the same burning rate at 2000 psia. Also, replacing the aluminum powder with magnesium powder and a 65/35 magnesium-aluminum alloy powder showed essentially no change in burning rate at 2000 psia.

(U) A new lot of UFAP (0.55.) was evaluated in the series where IPDI was varied from 45-55-65 equivalents. The burning rates respectively, were 3.28, 3.39 and 3.51 in/sec at 2000 psia. The data indicate a correlation of burning rate either with modulus, IPDI or unreacted hydroxy level. No firm conclusion can be made with the limited data currently available.

(U) A small amount of a new lot of crystalline Fe_2O_3 received from NWC was evaluated along with a new 10-lb lot purchased from Frank C. Davis Company which was used as received and after grinding for two hours. No changes in burning rate were observed with the unground samples, but the ground material showed a 5% increase.

(U) A series of propellant "B" batches were evaluated in which the catocene level and oxidizer ratios were varied. The desired burning rate was exceeded at a catocene level of 4%. The oxidizer modifications at 3% catocene fell short of the burning rate goal.

(U) Safety tests revealed that the propellant "B" formulation containing 4% catocene and having a 72 Shore "A" hardness, has high friction sensitivity, whereas a batch containing 3% catocene and having a 30 Shore "A" hardness, showed improved stability to friction. It has not been assessed whether hardness or catocene level was responsible for the high friction sensitivity.

(U) A DPT specimen utilizing the 434-4 liner reported in the first quarterly report was tested after being aged one month at 135°F. The results showed that the liner-propellant bond remained excellent. It is planned to use the 434-4 liner for this program and no further liner work is planned.

TECHNICAL DISCUSSION

GENERAL APPROACH

(U) The principal effort during this report period was directed toward increasing the potlife and burning rate of propellant "A". Increased potlife is necessary in order to successfully scale-up the propellant batch size and cast the requisite number of grains to fulfill the contractural requirements. Particular emphasis was placed on improving processability by introducing various binder modifications.

(U) Several attempts were made to extend potlife by inhibiting isocyanate groups with a reversible blocking agent and by using epoxides alone or in combination with the diisocyanate IPDI. These epoxides ranged from dicycloaliphatic to dialiphatic types to determine the effect of structural changes. As part of the isocyanate-inhibition approach, dimerized TDI was prepared and purified prior to incorporation into propellant. It was hoped that reversion back to monomer would be slow enough to yield potlife advantages.

(U) IDP was used in place of Oronite 6 to take advantage of its lower viscosity as a means to lower the propellant viscosity. Also, the IPDI concentration was reduced for an optimum balance between potlife and mechanical properties. Other approaches evaluated for improved processability included increasing the Oronite 6 at the expense of R-45M, further optimization of the bonding and wetting agents, and modification of the R-45M prepolymer.

(U) The burning rates of both propellants "A" and "B" were maximized, commensurate with good processability and minimum propellant hazard.

by improving the burning rate catalyst, using finer oxidizer blends, and extending the mix cycle. Other approaches consisted of replacing aluminum powder with magnesium powder and partially replacing the plasticizer with a fluorocarbon plasticizer which had given burning rate increases in other propellants.

(U) Principally, the work to increase burning rates was directed toward achieving the 3.5 in/sec burning rate goal at 2000 psia for propellant "A", but, as was previously demonstrated, improvements in the ballistic properties of "A" could be readily adapted to meeting propellant "B" ballistic requirements. Consequently, formulations for propellant "B" were evolved by making minor modifications in the "A" formulations.

PROCESSING AND MECHANICAL PROPERTIES STUDY

(U) Previous unsuccessful efforts to increase the potlife of propellant "A" were based on methods to block the isocyanate reactivity either by reaction with phenol or by dimerization with the dimerization catalyst trioctylphosphine oxide (TOPO). Failure to achieve the desired potlife increase for the first approach was attributed to the difficulty of the aliphatic diisocyanate IPDI to react with phenol in the absence of a catalyst whereas the second approach did not work probably due to inability of IPDI to dimerize. On the other hand, aromatic diisocyanates such as TDI readily react with hydroxy bearing compounds such as phenol; also are readily isolable as stable dimers. Consequently, these two blocking techniques were again attempted using TDI in place of IPDI. The method used in this case was to combine the TDI with the blocking or dimerizing agent 48 hrs. prior to incorporation into the propellant. The blocking agent used was acetylacetone in place of phenol to provide a more readily reversible blocked isocyanate, and the dimerization catalyst used was TOPO at a level equal to fifty percent of the isocyanate level. The appearance of finely divided solid material was taken as evidence in both cases for the formation of the desired modified isocyanate and no further analysis was attempted. These modified TDI mixtures were then incorporated into the standard R-45M propellant formulation, but yielded very short potlives. Apparently the blocking and dimerizing reactions did not proceed far enough before incorporation into propellant. No mechanical or ballistic properties were measured on these propellants. Also, no further attempts at forming blocked isocyanates were made during this report period.

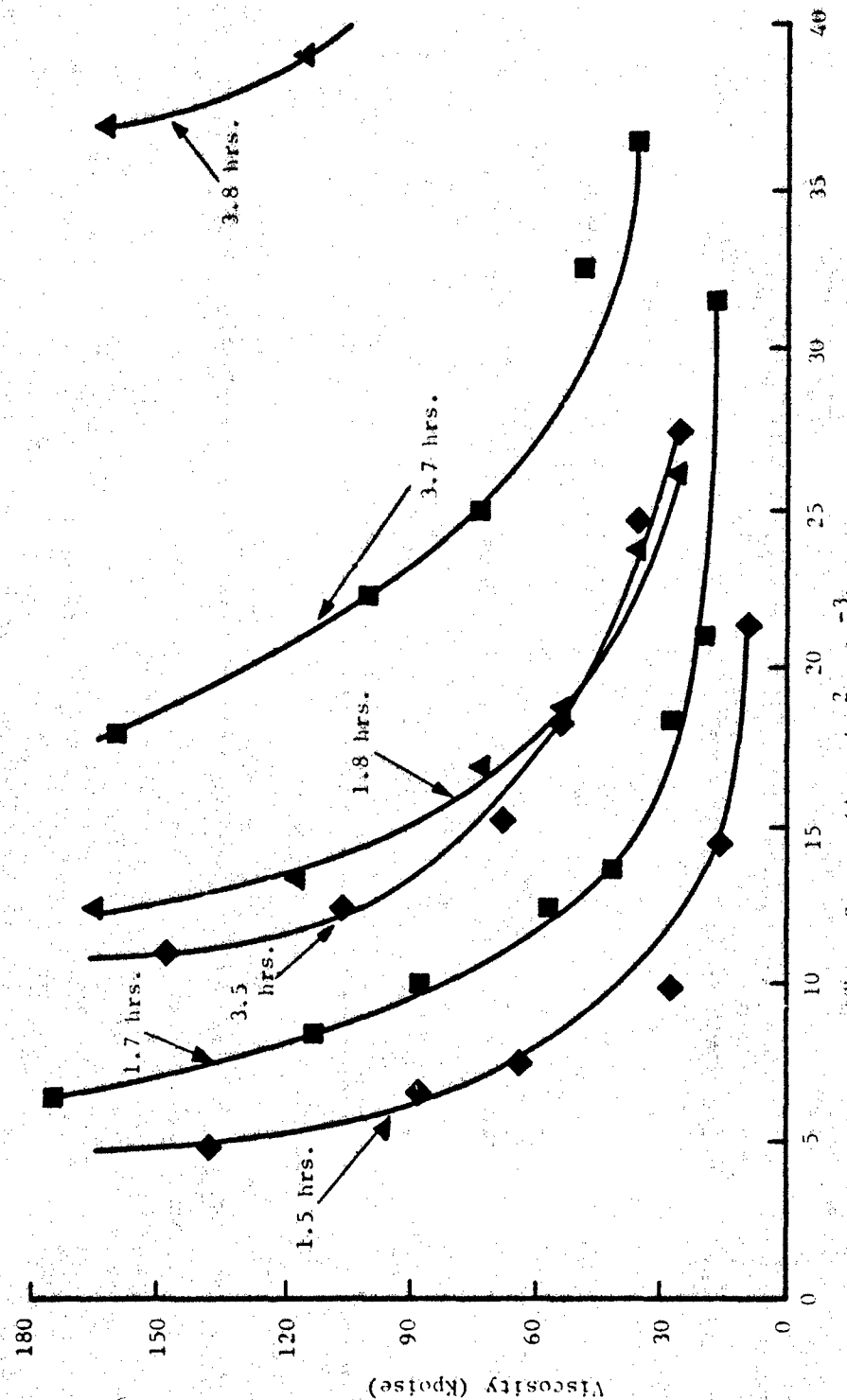
(U) A final evaluation of the value of the dimer approach to increased potlife was made (Table 1, #AK-7591-17, -24A,-24B) by using the purified dimer of TDI as the curing agent in place of IPDI.

The dimer was formed by blending 1 mole of TDI in 500 ml toluene with 5 drops of tri-n-butylphosphine as the catalyst. The mixture was stirred overnight and the resulting solid was collected by filtration and washed well with hexane. The vacuum dried material had an m.p. of 154-156°C identical to the literature value. The I.R. spectrum was also in accord with the expected dimerized structure, giving a strong isocyanate absorption at 2280 cm^{-1} and a strong urea-like carbonyl absorption at 1772 cm^{-1}. The propellants made with this material gave reasonable potlives, but cured rapidly to a hard, brittle material with apparently poor mechanical properties. Since no great advantage in potlife was demonstrated (Figure 1, #AK7591-24A), and since the resultant mechanical properties were poor, no further work was done with this material.

(U) Epoxide and imine curing agents were evaluated in this case to see if the catalytic nature of the propellant solids could promote either the imine- or epoxy-hydroxyl reaction. The diimine BISA was used first, but failed to give any potlife. It cured the propellant rapidly to a hard, brittle mass. The use of the diepoxide vinylcyclohexene dioxide (ERL-4206), which was evaluated next, resulted in a 4 hour potlife at 135°F; the material gelled but failed to develop a satisfactory elastomeric cure. A reasonable cure was achieved, however, by replacing one third of the ERL-4206 with IPDI (Table 1, #AK7591-13). Although this shortened the potlife to two hours, it was still estimated to be two times that with IPDI as the sole curing agent. Potlife was taken as time to 50 kpoise at 120°F and 10,000 dynes/cm^2 shear stress.

(U) Since ERL-4206 contains two kinds of epoxides, aliphatic and cycloaliphatic, it was of great interest to see if they behaved differently from each other in their reactivity with R-45. The dicycloaliphatic epoxide ERL-4221 was therefore evaluated in another batch of propellant, resulting in virtually no potlife and a rapid cure to a hard brittle propellant practically identical to that obtained with the imine BISA. The indication is that there is a vast difference in reactivity between the two types of epoxides, but it is not known whether or not the two types undergo different reactions.

(U) The use of various epoxides in combination with IPDI as a means of extending potlife but still achieving an adequately cured propellant was evaluated further. In a series of three batches, (Table 1, #AK7591-24C, -24D, -24E) the IPDI was formulated at 35 eq. alone and in combination with 65 equivalents each of resorcinoldiglycidyl ether (CIBA ERE 1359) and vinylcyclohexene dioxide (Union Carbide ERL-4206). The purpose of using IPDI alone was to determine whether or not the cures seen with the IPDI-epoxy combinations were due only to the IPDI. The shore hardness data indicate that the epoxides used do contribute to the cure. Initial viscosity as well as buildup with time is greater when 4206 and IPDI are mixed than when IPDI was used alone (Figure 1,



(U) FIG. 1. Viscosity vs. Shear Stress at 120°F and Two Time Intervals. The Time Noted on Curves Denotes Time After IPDI Addition. Curves include (1) ■ Biner TDI (AK7591-24A), (2) ▲ IPDI 35 eq./4206 eq. (AK7591-24E) and (3) ◆ IPDI 35 eq. (AK7591-24C).

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(C) TABLE 1. Processing and Mechanical Property Variations
Propellant "A"

Batch No. AK-	AP Coarse Fraction, μ	Curing Agent	Ballistic Prop. r_{2000}, in/sec	Ballistic Prop. Pressure Exponent, n	Mechanical Prop. m', psi	Mechanical Prop. m'	Mechanical Prop. E_o, psi	120°F Pot-Life, hr. to 50 K-Poise at Infinite Shear	Shore "A" Hardness (5 days at 135°F)
7591-11[a]	3	IPDI (70)	3.25	0.78	89.5	19.6	516	--	59[b]
7591-13[a]	3	IPDI (35)/4206 (65)	2.98	0.76	43.7	21.9	282	--	12[b]
7591-17	3	TDI-D[f] (70)	--	--		Poor		--	50
7591-24A	8	TDI-D[f] (65)	--	--		Poor		5	38
7591-24B	8	TDI-D[f] (35)/4206 (65)	--	--	--	--	--	5	5
7591-24C	8	TDI-D[f] (35)	--	--	--	--	--	10	0
7591-24D	8	IPDI (35)/RDE (65)	--	--	--	--	--	6	25
7591-24E	8	IPDI (35)/4206 (65)	--	--	--	--	--	6	10
7565-71[d]	8[e]	IPDI (70)	3.05	0.76	129	18.3	760	4 (110°F)	71
7591-30[d]	5	IPDI (45)	3.28	0.80	55.1	33.8	243	8	18[c]
7591-32[d]	5	IPDI (55)	3.39	0.79	71.2	25.7	344	6	33[c]
7591-34[d]	5	IPDI (65)	3.51	0.78	120	14.2	935	5	65[c]

NOTE: All propellants contain 50% 0.4μ UFAP, 19% 3.8-8μ UFAP, 0.5% Fe_2O_3, 0.5% Silon-S, 15% R-45M/IPDI binder, except where noted, and mix cycles have been set at 4 hours.

a Contained IDP in place of Oronite 6.
b Cured 2 weeks at 135°F.
c Cured 6 days at 135°F.
d Contains 50% (0.55μ) UFAP.
e Increased to 20% of formulation.
f Dimerized TDI

#AK7591-24E,-24C). The ERE-1359 is representative of a dialiphatic epoxide and showed a higher initial viscosity than did the ERL-4206. This was probably due to poor miscibility of this diepoxide in the R-45/Oronite 6 mixture because of its aromatic nucleus. This point was checked out by substituting the diglycidyl ether of butanediol (RD-2) for ERL-4206 at two levels as a co-curing agent with IPDI. This epoxide (RD-2) in combination with IPDI unexpectedly shortened potlife drastically and increased the brittleness of the cured propellant. Since the epoxides have not provided significant improvements in processing and mechanical properties, they have been dropped from further consideration in these propellants.

(U) A batch was processed to determine whether or not replacement of Oronite 6 with IDP would give any viscosity benefits without adversely affecting mechanical properties. Although the complete replacement of Oronite 6 by IDP resulted in improved processing, this was offset by a reduction in both mechanical properties and burning rate of the cured propellant (Table 1, #AK7591-11, -13).

(U) Because of the long interim mix cycle and the coarse aluminum used in these propellants, it was desirable to know whether or not the aluminum was producing any side effects as a result of abrasion. The data (Table 2, #AK7591-26) indicate a slight lowering of the burning rate and a somewhat harder cure than is normally seen. If the data are significant, then a possible explanation for the lower burning rate could be less efficient breakup of the UFAP particles and the harder cure could be attributed to less reaction of the fresh aluminum surface with the hydroxyl groups thereby allowing more complete reaction with the isocyanate curing agent. The effect is small enough, though, to be considered as a normal variation for this propellant.

(U) Two batches were made in which the ballistic solids were lowered by one and two percent, respectively (Table 2, #AK5691-42 and -28). Significant improvements in processing and mechanical properties were seen, especially for the 83% solids formulation. The sacrifice in burning rate, however, was too great to give serious consideration to this approach to improving processing and mechanical properties.

(U) Since all of the hydroxyl groups on the R-45M were not being used in the polymerization due to the less than stoichiometric level of IPDI, the level of R-45M was reduced to the exact stoichiometry with the IPDI present, while the Oronite 6 was increased to make up the difference(Table 2, #AK7591-52). It was hoped that the processing and potlife would be significantly improved with the increased plasticizer level without adversely affecting other parameters. The data indicate that no significant benefits were realized, while the burning rate and mechanical properties were adversely affected. No further efforts in this direction are planned.

(U) The observation that residual R-45M on the outside of the can eventually formed a polymer led to the speculation that oxidative crosslinking may be slowly causing a viscosity increase in the R-45M being used. An identical lot of this R-45M was evaluated to see if any differences in processing were apparent (Table 2, #AK7591-56). Although the data show no significant differences, there was a noticeable improvement in the processing, indicating that the exposed can of R-45M had undergone a small amount of oxidative crosslinking. Precautions are being taken to minimize such deteriorations.

(U) A significant improvement in potlife was achieved by using R-45M modified to remove the inactive hydroxyl groups (Table 2, #AK7591-62, -76). A graphic illustration of the effect of modified R-45M (designated R-45M-25 in this case) on viscosity buildup at two different shear stresses is presented in Figure 2, where a comparison is made with an identical batch using unmodified R-45M. The changes in mechanical properties are not great when compared to formulations having the same binder composition using unmodified R-45M (Table 3, #AK7591-68, -70). Further evaluation of modified R-45M is in progress to optimize processing and mechanical properties.

(U) Since previous work had shown that 70 equivalents of IPDI still produced too hard a propellant (Table 1, #AK7365-71) and current work (Table 1, #AK-7591-24C) showed a significant increase in potlife by lowering IPDI content, a series of three batches were prepared (Table 1, #AK7591-30, -32, -34) varying the IPDI from 45-65 equivalents. Rotovisco data (Figure 3) were taken on these batches to determine the viscosity buildup at each IPDI level. The data indicate that an IPDI level of 55-60 equivalents is desirable from the standpoint of processing and mechanical properties.

(U) A final assessment of the importance and desired levels of DEO and TEA in propellant "A" was made in a three batch series (Table 2, #AK7591-36, -38, -40). The data clearly indicate that the presence of both TEA and DEO are both beneficial from a processing as well as a mechanical property standpoint and are adequate at the 5 and 10 equiv. level, respectively. When neither were used, the resulting propellant had a pasty appearance as well as a high initial viscosity. The initial viscosity came down as TEA and then TEA and DEO were added, producing a noticeably smoother and more castable propellant. The data also indicated that 65-70 equiv. IPDI is needed with the lowered DEO level. The progressively longer potlife is the result of lower initial viscosities.

PROPELLANT "A" BALLISTIC STUDY

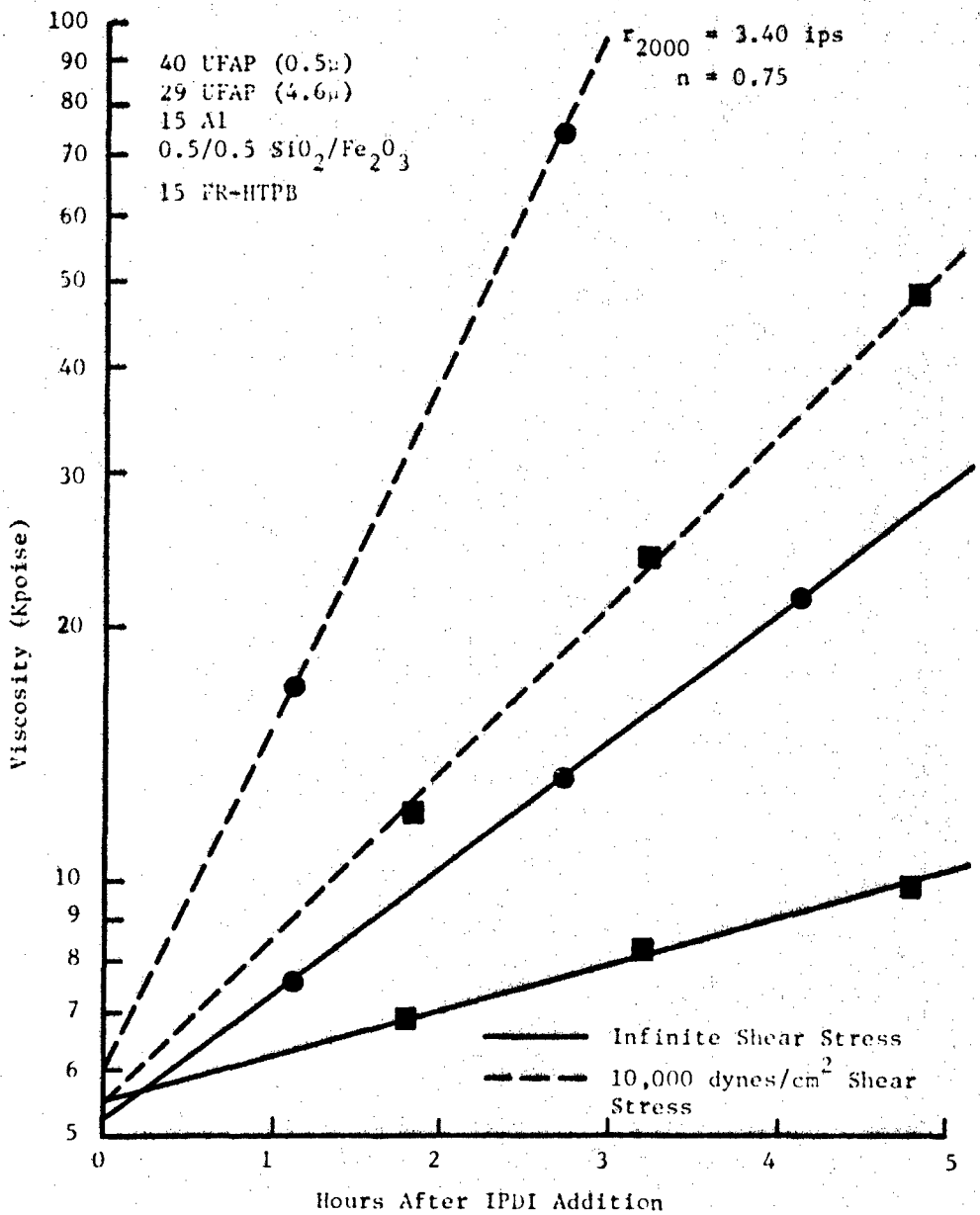
(U) An attempt was made to incorporate a fluorocarbon ester into propellant "A" and evaluate its effect on burning rate. This ester was prepared by condensing 216 gms (0.5 mole) of 1,1,9-trihydrohexadecafluoro-1-nonanol with 144 gms (1.0 mole) of 2-ethylhexanoic acid

(C) TABLE 2. Processing and Mechanical Property Variations
Propellant "A"

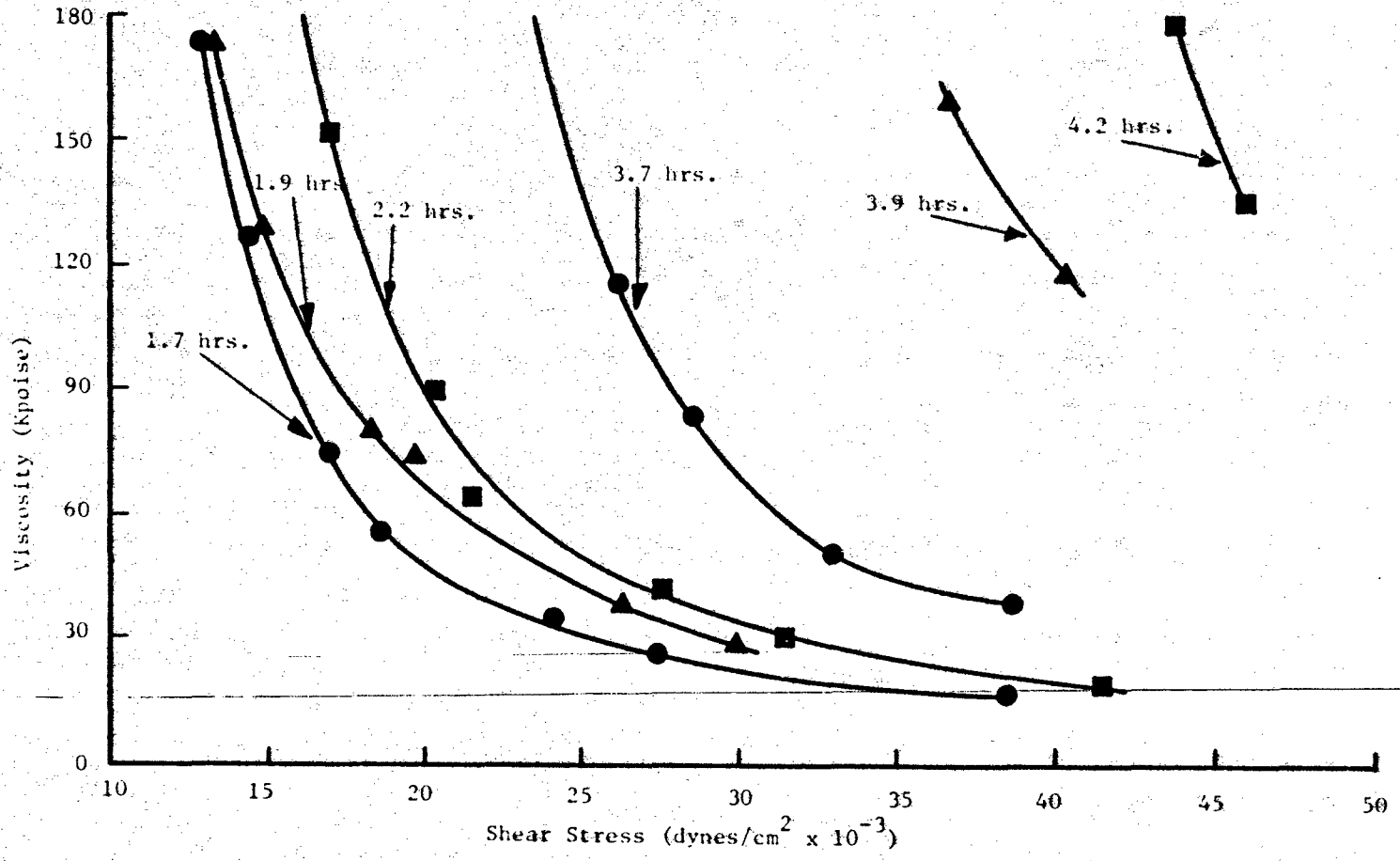
Batch # AK7591-	Binder			Ballistic Prop.		Mechanical Prop.			120°F Time, hrs. to 50 Kpoise at · Shear	Shore "A" 6 days, 135°F
	R-45M (Eq.)	DEO (Eq.)	Oronite-6 (")	r_{2000} in./sec.	Pressure Exponent n	σ_m^* psi	ϵ_m^* %	E_o^* psi		
26[a]	75	20	4.80	3.37	0.76	153	19.9	847	6.5	75
28[b]	75	20	5.45	3.07	0.72	147	30.5	563	9.0	67
42[c]	75	20	5.80	3.25	0.76	129	21.8	660	7.0	68
36[d]	100	--	4.80	3.52	0.76	81.6	18.5	474	4.5	52
38	95	--	4.80	3.40	0.74	92.2	19.2	507	5.0	56
40	85	10	4.80	3.48	0.78	118	19.4	674	6.0	64
52	55	10	8.00	3.32	0.78	149	11.0	1357	7.0	78
56[e]	85	10	4.80	3.40	0.78	107	18.3	635	6.5	62
62	(85)[f]	10	4.80	3.40	0.78	50.2	30.9	242	9.5	18
76	(85)[g]	10	4.50	3.40	0.75	52.1	29.1	251	13.5	28

NOTE: All batches contain (50%) 0.55μ UFAP, (19%) 5μ UFAP, 15% Al H-95, and 15% binder with TEA and IPDI held constant at (5 eq) and (70 eq), respectively, unless otherwise noted. Mix cycles are 4 hours.

a Al H-95 added one half hour prior to IPDI addition.
b Total solids decreased two percent at expense of Al H-95.
c Total solids decreased one percent lowering Al H-95 two percent, increasing 5μ UFAP one percent.
d No TEA.
e Used unopened can of the same lot of R-45M used in all other batches.
f Modified R-45M calculated as unmodified R-45M and designated R-45M-50.
g Same as (f) with the designation changed to R-45M-25 and IPDI lowered to 65 eq.; ground crystalline Fe_2O_3 was used, and oxidizer changed to (40%) 0.55μ UFAP and (29%) 5μ UFAP.



(C) FIG. 2. Viscosity Buildup at 120°F on Propellant "A" with (1) ● Regular R-45M (AK7591-70) and (2) ■ Modified R-45M (AK7591-76).



(U) FIG. 3. Viscosity vs. Shear Stress at 120°F and Two Time Intervals. The Time Noted on Curves Denotes Time After IPDI Addition. Curves Include (1) ● IPDI 45 eq. (AK7591-30), (2) ▲ IPSI 55 eq. (AK7591-32) and (3) ■ IPDI 65 eq. (AK7591-34)

catalyzed with one gram of conc. H_2SO_4 all in 250 ml of toluene. The solution was refluxed for 6 hrs. in which time nine ml of water was azeotroped off by the toluene signifying completion of the reaction. The toluene solution was washed twice with distilled water dried over anhydrous $MgSO_4$ and filtered to remove drying agent. The toluene was removed under vacuum on a rotary film evaporator and the resultant oil was distilled under vacuum to yield ~200 gms of the desired ester boiling at 107-109°C/3mm Hg. Only half of the Oronite 6 was replaced with this material, but it made the propellant difficult to process resulting in a brittle propellant which could not be tested for burning rate. Since it appears that significant amounts of fluorine cannot be incorporated into this propellant system without severely degrading processing and mechanical properties, no further effort in this direction is planned.

(U) A finely ground ferrocene-formaldehyde polymer was evaluated at the two percent level to see if its behavior was like that of the liquid ferrocene derivatives, i.e., show a nearly linear increase in burning rate with increase in ferrocene content (Table 4, #AK7591-50). Not only did it not significantly increase burning rate, but it severely curtailed processability and promoted a very rapid cure. A possible explanation for its poor behavior as a burning rate catalyst is its high polymeric nature which reduces its ability to interact with the NH_4ClO_4, contrary to the low molecular weight liquid and solid ferrocene derivatives, such as catocene and n-butyl ferrocene.

(U) Significant increases in the burning rate of propellant "A", however, were achieved during this report period (Table 3, #AK7591-93, -75). The first ballistic improvement yielded a burning rate at 2000 psia approximately seven percent higher than the control batch and was achieved by replacing the pigment grade iron oxide (C. K. Williams, Lot #RY2196) with a red crystalline iron oxide (F. C. Davis Company), a sample of which was provided by the Naval Weapons Center. No other effects, adverse or otherwise, were observed with this new iron oxide. Therefore, all future propellant "A" formulations are being planned with it. This increase brought to three the number of independently achieved burning rate increases to date, the other two being achieved by (1) replacing the 8u MA AP with 3u UFAP and (2) increasing the length of the mix cycle, the results of which were reported in the previous quarterly report. All three factors were combined into one propellant to yield a solid strand burning rate of 3.62 in/sec at 2000 psia which exceeded the minimum program goal for propellant "A".* An identical batch prepared to confirm this burning rate (Table 3, AK7591-19), yielded a burning rate of 3.4 in/sec. at 2000 psia. The reason for the lower rate is uncertain, but may be a result of different testing methods used. In the former batch, the strands used were exactly 1' long and were fired in a closed bomb. The repeated batch was tested with standard 5" strands.

* See Table 3 (AK7591-1)

(C) TABLE 3. Ballistic Variations Propellant "A"

Batch No. AK-	Formulation Variables: BR Catalyst at 1	Metal Powder at 15	Coarse AP at 19	Mix Cycle, hrs.	r_{2000} in./sec.
7365-57[a]	50/50 Silon S/Fe_2O_3	Al-H95	MA AP (10..)	1.1	2.90
7365-75	50/50 Silon S/Fe_2O_3	Al-H95	UFAP (3..)	1.3	3.15
7365-93	50/50 Silon S/Fe_2O_3[b]	Al-H95	MA AP (10..)	1.5	3.20
7591-5A	Fe_2O_3	Al-H95	UFAP (3..)	2.5	3.00[c]
7591-5B	50/50 Silon S/Fe_2O_3[b]	65/35 Mg/Al alloy	UFAP (3..)	2.5	3.15[c]
7591-5C	50/50 Silon S/Fe_2O_3[b]	Mg	UFAP (3..)	2.5	3.14[c]
7591-5D	50/50 Silon S/Fe_2O_3[b]	Al Class II	UFAP (3..)	2.5	3.14[c]
7591-1	50/50 Silon S/Fe_2O_3[b]	Al-H95	UFAP (3..)	4.5	3.62[c]
7591-19	50/50 Silon S/Fe_2O_3[b]	Al-H95	UFAP (3..)	4.5	3.40

NOTE: All formulations contained 50% UFAP (0.4μ) and 15% R-45-IPDI binder.

[a] Control batch.
[b] Red crystalline iron oxide.
[c] Small solid strands fired in a closed bomb.

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(U) It was of interest at this time to learn if the same burning rate achieved with the 50/50 mixture of Silon S and the red crystalline iron oxide could be achieved or surpassed by using this new iron oxide as the sole burning rate catalyst at the same total percent. The rate achieved (Table 3, #AK7591-5A) did not equal the rate (Table 3, #AK7365-93) achieved by the 50/50 combination of Silon S and the same crystalline iron oxide. This result indicates that both catalysts are needed in combination to achieve the optimum burning rate.

(U) A new lot of coated submicron UFAP was prepared and MSA analysis revealed the average particle size to be 0.55μ. Three batches were prepared during this report period using this material (Table 1, #AK7591-30, -32, -34). The burning rate data indicate that the new 0.55μ UFAP achieves the burning rate of 3.5 in/sec at 2000 psia. The interesting fact, however, is the variation of this burning rate among the three batches. There seems to be a correlation of burning rate either with urethane level, unreacted hydroxy level or propellant hardness. It is not known at this time which, if either, is responsible for the variation in burning rate, since additional batches did not substantiate this trend.

(U) One other ballistic modification that yielded no increase in burning rate consisted in the replacement of the aluminum with magnesium and a 65/35 magnesium/aluminum alloy powder. A comparison of the burning rates (Table 3, #AK7591-5B, -5C, -5D) of these with a batch containing a conventional aluminum powder (Grade II) revealed that the change from aluminum to magnesium had no effect one way or the other on the burning rate. The failure, however, of magnesium to elevate the burning rate was not too surprising, since burning rate increases observed with this metal powder were in propellants containing varying amounts of fluorocarbons. The magnesium powder did not adversely affect processing or cure.

(U) A small sample of crystalline Fe_2O_3 (Lot 15) from NWC was evaluated along with a 10 lb. lot of crystalline Fe_2O_3 recently purchased from Frank C. Davis Company. No differences in burning rate were observed (Table 4, #AK7591-48, -58). The batch containing the (Lot 15) Fe_2O_3 did process somewhat better, however. A significant increase in burning rate (Table 4, #AK7591-60) was achieved, though, by grinding a portion of the crystalline Fe_2O_3 from the 10 lb. lot for two hours in Freon using Al_2O_3 1/4" cylinders for grinding, and a mechanical paint shaker to provide agitation. The resulting powder was obviously much finer than the unground material and was much redder in appearance. The average particle size has not yet been determined. The finer Fe_2O_3 also appeared to function as a better cure catalyst since the data show a shorter potlife for this batch.

(U) Since the ground Fe_2O_3 gave a faster burning rate, it became desirable to determine if the concentration of 0.55μ UFAP may be reduced and still meet the 3.5 in/sec. burning rate goal at 2000 psia. The data

(Table 4, #AK7591-68, -70) indicate that a drop of 5 wt. % in this fine oxidizer very nearly meets the desired burning rate. Also, the processability improves very rapidly as the level of 0.55μ UFAP is lowered. The binder was altered for these batches to provide a softer propellant, which explains the lower tensile strength and higher elongation values.

PROPELLANT "B" BALLISTIC STUDY

(U) Utilizing the information gained in developing propellant "A", a batch of propellant "B" was prepared (Table 5, #AK7591-54) that exceeded the 7.0 in/sec. burning rate goal at 2000 psia using four percent catocene rather than the five percent Hycat 6 that was required to reach the same rate on the previous program. Additionally, better mechanical properties were realized than before, but the Shore "A" hardness was too high and the potlife was not as long as desired. It is expected that the potlife will be significantly extended by the use of modified R-45M.

(U) Having achieved the desired burning rate with four percent catocene, it became of interest to see how high a burning rate could be achieved at three percent catocene level. Three batches were prepared (Table 5, #AK7591-66, -72, -74) in which the 0.55μ UFAP was varied from 50% to 40% in combination with MA-AP and 5μ UFAP. The data show that the catalyst level is significantly more effective in raising the burning rate than increasing the fine oxidizer content. The final choice for ballistic properties appears to be between 3.5 - 4% catocene and 40 - 45% 0.5μ UFAP. The choice will be made based on overall properties within 30 days.

PROPELLANT "B" HAZARD TESTS

(U) Safety data acquired on the formulation containing four percent catocene indicate this propellant is type 5D due to high friction sensitivity. However, at three percent catocene the friction sensitivity was quite low indicating a less hazardous propellant. Unfortunately, the more sensitive propellant also had a high shore hardness while the less sensitive one had a low hardness reading, and since friction sensitivity tends to go up with propellant hardness it is not known whether high catocene, high shore hardness or both are responsible for the increased friction sensitivity. This point is currently being investigated.

(C) TABLE 4 . Ballistic Variations Propellant "A"

Batch # AK7591-	Oxidizer		Ballistic Prop.		Mechanical Prop.			120°F Time, hrs. to 50 Kpoise at * Shear	Shore "A" 6 days, 135°F
	% UFAP (0.55μ)	% UFAP (5μ)	r_{2000} in./sec.	Pressure Exponent n	σ_m, psi	ϵ_m, %	E_o, psi		
50[a]	50.00	19.00	2.92	0.67	--	--	--	Too Viscous	68
48[b]	50.00	19.00	3.40	0.76	96.0	16.6	616	7.0	65
58[c]	50.00	19.00	3.40	0.77	118	19.8	670	6.5	66
60[d]	50.00	19.00	3.56	0.79	124	18.1	772	5.5	63
68[d,e]	45.00	24.00	3.47	0.77	73.2	25.1	344	6.5	37
70[d,e]	40.00	29.00	3.40	0.75	76.6	24.1	353	7.5	44

NOTE: All batches contain 69% AP, 15% H-95 Al, 0.5% Silon S, 0.5% Fe_2O_3, 15% binder [(0.2% aging stabilizers, (4.8%) Oronite 6, (85 eq.) R-45M, (10 eq.) DEO, (5 eq.) TEA, (70 eq.) IPDI] unless otherwise noted. Mix cycles are 4 hours.

a
b Contains 2% ferrocene formaldehyde polymer replacing 1.0% Al H95, 0.5% Silon S, 0.5% Fe_2O_3.
c Contains new lot crystalline Fe_2O_3 received from NWC.
d Contains new 10# lot of crystalline Fe_2O_3 purchased from Frank C. Davis Co.
e Contains new 10# lot of crystalline Fe_2O_3 ground 2 hrs. on a paint shaker.
Contains (65 eq.) IPDI, (4.5%) Oronite 6, (0.5%) aging stabilizers.

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(C) TABLE 5. Ballistic Variations Propellant "B"

Batch # AR7591-	Oxidizer			Ballistic Prop.		Mechanical Prop.			120°F Time, hrs. to 50 Kpoise at 1 Shear	Shore "A" 6 days, 135°F
	UFAP (0.35μ)	UFAP (5μ)	MAAP (10μ)	r_{2000} in./sec.	Pressure Exponent n	m' psi	m' ~	E_o' psi		
34[a]	45.00	--	17.00	7.30	0.84	145	20.0	901	4.5	72
66	50.00	--	12.00	6.40	0.85	74.6	22.6	433	Too Viscous	45
72	45.00	17.00	--	6.55	0.85	60.8	28.4	307	4.0	32
74	40.00	22.00	--	6.40	0.84	53.1	30.3	240	4.5	30

NOTE: All batches contain (62%) Fine AP, (8%) Ung PAP, (15%) Al H-95, (3%) Catocene, 12% Binder [(0.5%) aging stabilizers, (1.5%) Oronite 6, (85 eq.) R-45M, (10 eq.) DEO, (5 eq.) TEA, (65 eq.) IPDI] unless otherwise noted. Mix cycles are 4 hours.

[a] Contains (4%) Catocene, (0.2%) Aging Stabilizers, (0.8%) Oronite 6, (75 eq.) R-45M, (20 eq.) DEO, (5 eq.) TEA, (70 eq.) IPDI.

HAZARDS TEST DATA

Propellant "B"

Batch No.	AK7591-54 (See Table 5)	AK7591-74 (See Table 5)
Bureau of Mines Impact		
50% Fire Point	11 cm/2 kgm	--
Friction (Rotary)		
Gms load/RPM	400/3000	2000/3000
DTA		
Onset of Exotherm	328°F	--
Exothermic Peak	380°F	--
Autoignition	423°F	--

LINER AGING STUDY

(C) In the previous report period several liners were evaluated with a 10- x 86" solids scale-up batch of propellant "A". A liner formulation designated 434-4 exhibited good bonding to this formulation when tested in a DPT specimen. This liner was prepared by precuring it for four hours at 135°F prior to casting in the propellant. This resulted in a DPT specimen that exhibited cohesive failure in the propellant. On the other hand this same liner when fully cured prior to propellant casting resulted in an adhesive failure. The effect of storage at 135°F on the bond strength was determined. The results as shown below indicate that the liner/propellant bond remained excellent after aging:

EFFECT OF LINER/PROPELLANT BOND
AFTER 1 MONTH STORAGE AT 135°F

Propellant "A"

Liner	Time Aged at 135°F	Tensile, psi	Type of Break
434-4	Initial	92.8	Cohesive in propellant
434-4	1 month	112.5	Cohesive in propellant

This liner appears to be satisfactory, and the plans are to use it as the liner for the propellant grains that are to be prepared and delivered to Naval Weapons Center as per contractual requirements.

WORK PLANNED FOR NEXT REPORT PERIOD

(U) The final selection of the two final candidate propellants, "A" and "B", will be made at the beginning of the next report period. Processing, mechanical and ballistic properties characterizations of the two propellants will be made as well as hazard classification for both the uncured and cured states. Preliminary aging studies will be initiated concurrently with the preparation and delivery of ten 1.8 in. by 6.25 in. long grains of each of the two candidates. Also, the preparation of four 5 in. O.D. by 15 in. long grains of each formulation will be initiated.

Glossary of Terms and Abbreviations

Agerite White	Antioxidant
AO-2246	Antioxidant
AP	Ammonium Perchlorate
BDB	Aerojet proprietary coating agent
BRA-99	Aerojet proprietary combustion catalyst
BRA-101	Aerojet proprietary combustion catalyst
CTPB	Carboxy terminated polybutadiene
DEO	Hydroxy functional wetting agent
DOA	Dioctyladipate
EDB	Aerojet proprietary fuel component
ERL-4205	Bis(2-3-epoxycyclopentyl)ether
ERL-4221	3,4-Epoxycyclohexylmethyl-(3,4-epoxy) cyclohexane carboxylate
FC-155	Aerojet proprietary fuel component
Freon-113	1,1,2 Trifluoro-1,2,2 Trifluoro-1,2,2 Trichloroethane
HC-434	Carboxy-terminated polybutadiene (Thiokol Chemical Co.)
HDI	Hexamethylene diisocyanate
HTPB	Hydroxy terminated polybutadiene
Hycat-6	A non-volatile liquid ferrocene derivative
IDP	Isodecyl pelargonate plasticizer
IPDI	Isophorone diisocyanate
Isonol	Phosphorous containing polyol
MA	Mikro-atomizer ground ammonium perchlorate
MSA	Mine Safety Appliances Co., particle size measuring apparatus, a liquid sedimentation technique
nBF	n-Butylferrocene
P-33	Thermal carbon black
PAP	Porous Ammonium Perchlorate
Plastinox 711	Antioxidant
R-45M	Free radical initiated HTPB
Refrasil	Silica Fiber
SS-AP	Slow-speed mikro-pulverized ground ammonium perchlorate

Glossary of Terms and Abbreviations (cont'd)

SURFAC OS	Carboxy functional wetting agent
TEA	Triethanol amine
TEHOS	2-Ethylhexylorthosilicate
Thixcin E	Modified 1-hydroxy stearin
UFAP	Ultra-fine ammonium perchlorate (<5μ)
VEM	Vibro-energy mill

ABSTRACT CARD

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~~Second Quarterly Progress Report, 1 August - 31 October 1970, (U)~~

Quarterly Progress Report 1 Aug - 31 Oct 70

R. L. Lou A. Katzakian

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KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Solid Propellant						
HTPB						
Ultrafine AP						
Porous AP						
High Burning Rate						
High Slope						
Ferrocene						

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